

# **INTERNAL REPORT**

## **BIOTREATABILITY OF A SITE SOIL CONTAMINATED WITH XYLENE AND DIOCTYL PHTHALATE**

**Don H. Kampbell, USEPA/RSKERL**

**Dennis D. Fine and Jerry W. Anderson  
ManTech Environmental Technology**

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## **Introduction**

A large number of organic compounds can be mineralized or transformed through microbiological processes. Degradation rates vary for different compounds and are influenced by variables such as concentration, nutrients, moisture, aeration, temperature, degree of acclimation, plus many others. In situ biodegradation as a cleansing technology has the attribute of being a process of nature.

A site was used for over 40 years to manufacture vinyl wallcovering. Chemical spillage and waste disposal has contaminated portions of the site with metals and various organic chemicals, predominately xylene and dioctyl phthalate.

Treatability studies were conducted using laboratory techniques on vertical profile core samples obtained from the site. The objective of the study was to determine whether in-situ biodegradation is a feasible alternative for remediation of the site.

## **Experimental Methods**

Vertical profile core samples were collected to a depth of eight feet by Ken Tyson of Weston Consultants at the L.E. Carpenter site near Wharton, New Jersey on October 24-25, 1991. These locations were sampled: TS-01 = no contamination; TS-02 = moderate contamination; TS-03 = high contamination (Figure 1). The core samples representing three two-foot increments were placed in glass pint jars and shipped to our facility. The nine samples were analyzed by standard tests as shown in Table 1. A mixture of the three depth core samples from TS-03 location was extracted with methylene chloride for organic compounds identification by a gas

chromatography/mass spectrometer method. Total phthalate concentration was 1.7 percent. Oxygen and carbon dioxide were measured by removing aliquots of headspace gas from 160 ml. microcosm glass serum bottles containing ten percent by volume core material and capped with teflon-coated butyl rubber septa. Core material used for testing was separated from coarse sand and pebbles which were about  $\frac{1}{3}$  the total mass of core material.

Determination of the rate of biodegradation was done by adding 50 grams of air-dry Rubicon Sand soil to replicate 160 ml. serum bottles. The air-dry soil contained 2.8 percent moisture. Four treatments used were as follows: 1 = air-dry soil as a control; 2 = nine percent by weight water was added; 3 = above water + 1000 ul JP4 jet fuel fumes; 4 = above water + 600 ul xylene mixture vapor. The air-dry soil in each bottle contained 0.5 grams dioctyl phthalate. The microcosms were acclimated for two weeks then initial treatment sets were extracted with methylene chloride for analysis of dioctyl phthalate by a gas chromatography method. A second treatment set was extracted and analyzed 42 days later.

## Results and Discussion

Nutrient requirements most limiting to microbiological processes in soils are nitrogen and phosphorus. All nine of the core samples tested contained sufficient nitrogen and phosphorus (Table 1). Bacterial cell counts for a viable soil are usually in the  $10^7$  to  $10^8$  range. The site core samples had a total cell count indicative of vigorous bacterial activity. Dehydrogenase activity also indicated the presence of high viability and the absence of toxicological restraints.

Chemical analyses confirmed that the magnitude of phthalate contamination at the three different coring locations was low, moderate, and above moderate. Moisture contents of the core materials ranged from 10 to 32 percent. Previous soil microcosm studies in our laboratory has determined that moisture levels in this range do not restrict microbial activity.

Diethyl phthalate represented 95 percent of the contaminants in a mixture of core material (Table 2). Other components such as the volatile aromatic hydrocarbons may vary at adjacent locations and depths depending on losses by emissions, degradation, dissolution by soil water, and original concentrations.

Active microbiological processes typically involve oxygen and carbon dioxide especially under aerobic conditions. Data recorded in Table 3 was generated by core material microcosms for different 22°C incubation time periods. A definite response occurred for oxygen consumption and carbon dioxide generation for the contaminated core materials. The trend of the data indicated that the rate of carbon dioxide generation was very close to the rate of oxygen consumption.

An estimation of degradation rate of diethyl phthalate in soil was determined with Rubicon Sand soil microcosms as listed in Table 4. Biodegradation by the control was limited by lack of soil moisture. Water and xylene vapors when present accelerated the rate of diethyl phthalate biodegradation. The data indicated that about 0.1 gram diethyl phthalate per kilogram soil per day was biodegraded. If a mass balance process could logically be extrapolated from lab studies to actual field conditions to obtain less than 10 ppm phthalates, the total cleansing time period for

in-situ biodegradation would be 1440 days (3 years) and 4380 days (12 years) for moderate and greater than moderate locations at the field site, respectively.

### **Conclusion**

The soil microcosm laboratory studies showed that cored material contaminated with the L.E. Carpenter site waste was being naturally remediated. The rate of biodegradation was estimated to be 0.1 milligram dioctyl phthalate per day per kilogram soil. Actual field rates can vary by orders of magnitude dependent on controlling factors. The lab studies did determine that the waste contaminated core materials are biologically active.

A potential for in-situ bioremediation exists at the site. Possibilities exist to enhance the waste biotransformation process by installation of a bioventing treatment system. Development of a productive unit process would require further effort in lab studies, a pilot plant, literature search, and experienced personnel.

**Table 1 - Dioctyl Phthalate, Nitrogen, Phosphorus, DHA, and Bacteria Cell Count  
for Core Samples from L.E. Carpenter Site\***

<b>Sample</b>	<b>Depth (ft.)</b>	<b>AODC cells/gm X10<sup>8</sup></b>	<b>DHA formazan µg/gm</b>	<b>Dioctyl Phthalate mg/Kg</b>	<b>Nitrate &amp; Nitrite mg/Kg</b>	<b>Total Phosphorus mg/Kg</b>	<b>Total Kjeldahl Nitrogen mg/Kg</b>
TS-01-01	2-4	34	3.7	8	6.5	432	497
TS-01-02	4-6	15	2.2	7	7.8	428	298
TS-01-03	6-8	14	3.6	4	11.0	379	807
TS-02-01	2-4	25	21.0	144	8.5	1060	665
TS-02-02	4-6	30	58.0	139	1.2	941	1510
TS-02-03	6-8	53	31.0	20	32.0	731	848
TS-03-01	2-4	8	22.0	152	3.9	722	1510
TS-03-02	4-6	14	6.6	438	1.2	573	2030
TS-03-03	6-8	7	2.5	410	1.2	638	1980

**\*Analytical Methods**

- AODC- Wilson, J.T., J.F. McNabb, D.L. Balkwill, and W.C. Ghiorse.  
"Enumeration and Characterization of Bacteria Indigenous to a  
Shallow Water Table Aquifer." *Ground Water*, 21, 134-142 (1983).
- DHA- Din, L.F. "Standard Operating Procedure for Dehydrogenase Activity."  
RSKSOP-100, USEPA/RSKERL, Ada, OK (1990).
- Dioctyl phthalate- Soxhlet Extraction - EPA Method SW846  
Extract analysis - EPA Method 525 except FID was used instead  
of MS/DS
- Nitrogen & phosphorus- EPA Methods 351.1, 353.1, 365.4



**Table 2 - Relative Abundance of Major Components In a  
Mixture of Cored Material from TS-03 Location**

<b>Component</b>	<b>Relative Abundance</b>
Ethylbenzene	0.18
m+p-Xylene	1.10
o-Xylene	0.21
Decane	4.96
Trimethyldecane	4.01
Ethylmethylheptane	1.52
4,7-Dimethylundecane	0.74
Octafluoronaphthalene (I.S.)	1.00
Unknown Phthalate	0.09
Butyl 2-methylpropylphthalate	0.52
2-Ethylhexyldiphenyl phosphate	3.75
Di-(2-ethylhexyl) phthalate	0.79
Diisooctylphthalate	91.98
Bis(2-ethylhexyl)phthalate or Dioctyl phthalate	241.45

**Table 3 - Microcosm Headspace Oxygen and Carbon Dioxide\***

Sample	Time Period, Days							
	2	5	17	24	2	5	17	24
	Oxygen, %				Carbon Dioxide, %			
TS-01-01	20.8	19.9	21.2	18.9	0.68	0.62	1.10	1.8
TS-01-02	20.7	19.9	21.1	18.9	0.70	0.71	1.10	1.6
TS-01-03	21.0	20.0	21.2	19.2	0.65	0.61	0.94	1.6
TS-02-01	18.1	16.7	10.5	7.5	3.40	3.20	8.00	10.1
TS-02-02	12.2	9.1	1.4	1.1	6.30	7.60	14.00	15.2
TS-02-03	16.2	14.1	9.1	6.0	4.70	5.40	9.40	11.6
TS-03-01	18.3	17.2	14.3	11.2	3.30	3.10	5.80	7.6
TS-03-02	6.7	8.6	1.0	1.1	9.00	8.40	14.70	15.6
TS-03-03	7.8	5.4	2.4	1.4	9.20	11.00	14.10	16.0

**\*Analytical Procedure**

Newell, B. "Standard Operating Procedure for Headspace Analysis for Oxygen and Carbon Dioxide." RSKSOP-114, USEPA/RSKERL, Ada, OK (1991).

**Table 4 - Degradation of Dioctyl Phthalate by Rubicon Sand Microcosms During  
42 Days Incubation at 22°C**

<b>Microcosm</b>	<b>Amount Dioctyl Phthalate Utilized from 10 gm Added/kg Soil</b>
Air-dry Control	1.0
Water Added	4.3
Water + Nutrient solution + JP4 Added	4.4
Water + Nutrient solution + Xylene Added	5.5

## STORAGE TANK INVENTORY



L.E. CARPENTER AND CO.  
WHARTON, NEW JERSEY

O-core sample locations  
GENERAL SITE PLAN 10/9/91

**FIGURE 1**

DATE  
6/19/

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